

1 Process for Particulate Material

2

3 The present invention relates to a process for
4 preparing a particulate material based on recycled
5 paper waste sludge containing cellulose fibres,
6 china clay and calcium carbonate, and products
7 prepared therefrom.

8

9 There is an increasing industry in recycling waste
10 paper. Waste paper is increasingly collected
11 throughout the UK with intent to recycle it.
12 However, the term "waste paper" includes a myriad of
13 different types of paper-fibre materials, all of
14 which are provided by the public in the belief that
15 they are all 'recyclable' in the same way.

16

17 A number of the "waste papers" also contain
18 materials that have been added during the original
19 paper production and conversion processes, and these
20 must be removed to provide a clean fibre material

1 suitable for re-use in a paper machine. Two main
2 materials that need to be removed from many types of
3 waste papers are china clay, also known as kaolin,
4 and calcium carbonate. These are added to certain
5 paper products to make the paper opaque and to
6 improve its printing quality. Other materials that
7 commonly need to be removed from waste papers are
8 staples, clips, glues, plastic coatings, etc.

9
10 Large separated materials from waste papers are
11 generally disposed of directly as "trash". These
12 include plastics, plastic coating materials and
13 metal clips, etc. Fine separated materials are
14 carried in an aqueous suspension to an effluent
15 treatment process where they are removed by gravity
16 sedimentation, and then further dewatered to 40-60%
17 solids for disposal. This creates a biological
18 sludge, (which can also be mixed with a primary
19 sludge before the dewatering stage).

20
21 The "paper waste sludge solids" are currently simply
22 deposited in landfill sites or spread on land.
23 However, increasing environmental legislation
24 requires this depositing to be reduced.

25
26 Meanwhile, paper making processes generally also
27 create a paper-fibre based sludge waste product
28 which is also currently deposited in landfill sites
29 or spread on land.

30
31 A typical quantity of waste sludge from a paper
32 recycling and paper making facility can be 250

1 tonnes per day, representing a significant amount of
2 waste sludge material.

3

4 It is an object of the present invention to provide
5 a process for treating such waste material to form
6 an industrial useful product.

7

8 Thus, according to one aspect of the present
9 invention, there is provided a process for preparing
10 a particulate solid material comprising the steps
11 of:

12

13 (a) obtaining a paper-fibre waste solid material
14 having a ratio of china clay, or equivalent, to
15 chalk, or equivalent, greater than a pre-determined
16 minimum;

17

18 (b) treating the material to reduce the moisture
19 content and form a granular material; and

20

21 (c) calcining the granular material at a temperature
22 of approximately 1300°C or higher to provide a
23 particulate, 100% solids, material.

24

25 The paper-fibre waste solid material can be
26 provided by the non-hazardous waste material arising
27 from the recycling of waste paper and tissue. Such
28 material is generally in the form of sludge, having
29 a moisture content of over 45%, and commonly over
30 55%, 60%, or higher. Such sludge contains china
31 clay, calcium carbonate as well as the general
32 cellulose fibre content. Such material may also

1 include surplus biomass from biological effluent
2 treatment processes and water treatment processes
3 which produce sludge. Minor components may include
4 non-fibrous 'contraries' materials arising from
5 waste paper, including such items as polythene,
6 plastics, metal (in the form of wire, staples, paper
7 clips).

8
9 The term china clay or equivalent includes any form
10 of hydrated aluminium silicate, including kandites,
11 kaolins and the like.

12
13 The term chalk or equivalent includes any form of
14 calcium carbonate, which includes the forms of
15 limestone.

16
17 The sludge is preferably dewatered so as to produce
18 a sludge having an increased solids content.

19
20 Analysis of the china clay:chalk ratio is preferably
21 carried out prior to the dewatering of the waste
22 material.

23
24 One method of analysis is termed 'acid extraction'.
25 A sample of sludge cake of known dry solids is
26 treated with 10% acetic acid solution to dissolve
27 calcium carbonate. The remaining solids are
28 filtered out, washed, dried at 105°C and weighed.
29 The loss in weight determines the calcium carbonate
30 content of the dry solids content of the sludge
31 cake. The remaining solids are further heated to a
32 high temperature such as, but not limited to, 800°C,

1 to form an ash free from carbon which again is
2 weighed. The further loss in weight determines the
3 cellulose content of the sludge cake. The remaining
4 weight of ash determines the clay content. From
5 these calculated values the clay:chalk (calcium
6 carbonate) ratio is calculated.

7

8 By the acid extraction method, the pre-determined
9 minimum is approximately 0.2.

10

11 Another method of analysis is termed "ash/acid
12 extraction". For this, a weighed sample of dried
13 sludge cake is treated to 800°C to form an ash free
14 from carbon. The cooled sample is weighed and the
15 loss in weight determines the cellulose content plus
16 the carbon dioxide arising from the destruction of
17 the calcium carbonate. The cooled sample is then
18 treated with 10% acetic acid, filtered, washed,
19 dried and weighed. The further loss in weight
20 determines the calcium oxide content and the
21 remaining weight determines the clay content. From
22 these measured values the cellulose, calcium
23 carbonate and clay contents can be calculated.

24

25 By the ash/acid extraction method, the pre-
26 determined minimum is approximately 0.13.

27

28 Dewatering is a process well known in the art, as
29 are the process parameters for the pressing action.
30 Traditionally, a polyelectrolyte is added to a waste
31 material, which material can often have only a 7%
32 solids content, in order to agglomerate the very

1 fine waste material, commonly termed "fines", in the
2 process. The dewatering process increases the
3 solids content several fold, such as to a typical
4 solids content of 45%.

5
6 Where the china clay:chalk ratio is found to be less
7 than the pre-determined minimum in the waste
8 material, an embodiment of the present invention is
9 to add a conditioning material to the waste
10 material. The conditioning material is preferably
11 partly, substantially or wholly china clay, or at
12 least a china clay suspension, or another silicate
13 material having the same properties. A dispersing
14 agent could also added to the conditioning material
15 in order to maintain the china clay or similar
16 material in a suspended form, in a liquid host such
17 as water.

18
19 It has been found that by varying the conditioning
20 material content in the waste material, the
21 dewatering process can result in a material having
22 less solids content than, for example 45%, such
23 content being even 22% or lower.

24
25 In an embodiment of the present invention, the
26 conditioning material is added to the waste material
27 even when the china clay:chalk ratio is greater than
28 the pre-determined minimum, in order to effect the
29 properties of the material as treated thereafter.

30

1 The dewatering process provides a sludge material
2 having a solids content generally in the range 22-
3 55%.

4

5 In another embodiment of the present invention, the
6 ratio of silica and aluminium to natural fillers in
7 the paper sludge is based on the addition of the
8 conditioning material which uses silica and/or
9 aluminium to promote fluxing of the paper sludge.

10

11 The treatment step (b) of the present invention
12 could be carried out by operating on the paper-fibre
13 waste material in various ways, such as compression,
14 extrusion or the like, and/or a combination of such
15 processes. Extrusion through a die with apertures
16 produces lines of a material which either inherently
17 or by further processing produces a granulated
18 material. A granulating press could be used for
19 this step (b).

20

21 The treatment step (b) of the present invention
22 could also be provided by direct heat contact, such
23 as conduction. A heat transfer material could be
24 used such as steam, etc. in this regard. The action
25 of the heat treatment is to partly or substantially
26 'dry' the paper-fibre based material.

27

28 Such a heat treatment process could be carried out
29 with agitation, such as provided by a rotary
30 apparatus such as a rotary dryer. An inclined
31 rotary processor can reduce water content while
32 inducing a tumbling action against heated surfaces

1 at controlled temperatures. Preferably, any heat
2 treating apparatus allows for a wholly or
3 substantially continuous feed of starting material.
4 The process may involve recycling of material to
5 adjust the range of particle sizes of granular
6 material produced.

7
8 The treatment step is preferably carried out at a
9 raised temperature, preferably between 60-80°C,
10 although not limited thereto.

11
12 The treatment step could also be carried out under
13 an inert atmosphere. Such an inert atmosphere could
14 be provided by displacing air with steam, either by
15 direct injection or by evaporated water. Heat
16 treating the material in an inert atmosphere reduces
17 the moisture content and forms the material into
18 rounded granules approximately 3mm-30mm in size.

19
20 In another embodiment of the present invention, the
21 granular material formed by the treatment step could
22 be further granulated, that is further processed in
23 a granulator or the like to better form a more
24 regular, generally spherical, shaped solids
25 material.

26
27 The material formed by the treatment step preferably
28 has a solids content in the range of approximately
29 45-90% solids. It has been found that the moisture
30 content of the so-formed granular material affects
31 the size of the particulate material formed in the
32 subsequent calcining step.

1

2 The calcining of the granular material is adapted to
3 reduce the moisture in the material to zero. The
4 particulate material being formed also becomes
5 porous, either partly or substantially, by the
6 burning of the cellulose content of the waste
7 material.

8

9 The calcining also fuses the granular material, so
10 as to provide solid pellets.

11

12 Preferably, the granular material is calcined with
13 agitation, such as provided by a rotary apparatus,
14 an example is a high temperature rotary furnace,
15 such as a tube. The rotary action serves to provide
16 a more even evaporation of moisture and burning of
17 cellulose.

18

19 Preferably, the calcining temperature is greater
20 than 1300°C, possibly approximately 1320°C or higher.

21

22 In one embodiment of the present invention, the
23 calcined product is reduced, for example granulated
24 or refined or otherwise ground. With certain size
25 reduction, such as milling or the like, a particular
26 particle maximum size or range of sizes can be
27 achieved. For example, through milling the calcined
28 particulate material to have a maximum particle size
29 of 100µm, a fine particulate material is formed
30 which is useable as a cementitious material, for
31 example as a partial, substantial or complete
32 replacement or substitute for current cement

1 materials in building blocks such as 'breeze blocks'
2 and the like.

3
4 According to a second aspect of the present
5 invention, there is provided a particulate solid
6 material whenever prepared by a process as herein
7 described.

8
9 The particulate solid material preferably has a bulk
10 density of less than $1,000\text{kg/m}^3$, and generally in
11 the range 560 kg/m^3 to 800 kg/m^3 . The particulate
12 solid material is preferably in the form of an
13 aggregate. Preferably, the aggregate has a mean
14 particle size of between 3-15mm. The mean particle
15 size can be favoured based on the moisture content
16 of the material made by the treatment step. The use
17 of a drier material provides a smaller mean particle
18 size, whereas the use of wetter material provides a
19 larger mean particle size.

20
21 The particulate solid material may be usable in a
22 number of industrial applications, including as a
23 light weight aggregate for making cementitious,
24 concrete or other building blocks, or as a
25 replacement or filler material in other building
26 applications. The material is also 'eco-friendly'.
27 Examples of the present invention will now be
28 described by way of example only.

29
30 Example 1
31

1 The starting material was provided from waste from
2 paper recycling in the form of raw separated solids
3 with water or sludge from biological or chemical
4 treatment of the separated solids. The starting
5 material included china clay, calcium carbonate,
6 cellulose fibres and a water content of
7 approximately 93%.

8
9 The material was analysed by the acid extraction
10 procedure to determine the ratio of clay:chalk and
11 to further determine the amount of conditioning
12 material to be added to bring the clay:chalk ratio
13 up to 0.2.

14
15 The conditioning material may include clay or chalk
16 and is prepared as a suspension in water at a
17 suitable concentration to facilitate pumping.

18
19 The material was fed to dewatering equipment with
20 conditioning material injected into the feed
21 pipeline. Dewatered material was collected from the
22 dewatering equipment with water content varying from
23 55% to 80%.

24
25 The dewatered material was fed at 10-15 kg/h into a
26 drier where heat transfer by contact with hot
27 surfaces evaporated water, formed the material into
28 rounded granules and provided an inert atmosphere.
29 The rounded granules were from 3-30mm in size, with
30 occasional granules up to 50mm in size, with a water
31 content of approximately 50%.

32

1 The dried material was fed at approximately 15 kg/h
2 into a calciner comprising a rotating tube lined
3 with refractory material and equipped with a propane
4 burner. The calciner was inclined so that material
5 passed counter current to the gas flow, passed
6 through a hot zone at 1300°C-1350°C before being
7 discharged into a receiving tray. The residence
8 time in the calciner was approximately 15-20 minutes
9 and the final processed material was hard to the
10 touch, light brown with a speckled surface in
11 appearance and produced a clear sound when dropped
12 onto a hard surface.

13

14 Example 2

15

16 The starting material was provided from waste from
17 paper recycling in the form of raw separated solids
18 with water or sludge from biological or chemical
19 treatment of the separated solids. The starting
20 material included china clay, calcium carbonate,
21 cellulose fibres and a water content of
22 approximately 93%.

23

24 The material was analysed by the acid extraction
25 procedure to determine the ratio of clay:chalk and
26 to further determine the amount of conditioning
27 material required to bring the clay:chalk ratio to
28 >0.2.

29

30 The conditioning material may include clay or chalk
31 and in this example was presented as a filter cake

1 which was prepared as a suspension in water at a
2 suitable concentration to facilitate pumping.

3
4 The material was fed to dewatering equipment with
5 the conditioning material injected into the feed
6 pipeline. Dewatered material was collected with
7 water content varying from between 40% and 45%.

8
9 The dewatered material was fed to a granulating
10 press fitted with an extrusion plate thus producing
11 extruded particles of approximately 20mm diameter
12 having a cylindrical form.

13
14 The extruded particles were transferred to a
15 granulating table comprising an inclined surface
16 with a rim to retain material, and inclined at an
17 angle so that rotation of the table produced rounded
18 particles. Small quantities of water were sprayed
19 onto the particles to assist with rounding of them.
20 Undersized and fine particles were separated by
21 screening and were returned to the granulating press
22 to be reformed.

23
24 The rounded particles were fed at a rate up to
25 approximately 35 kg/h into a calciner comprising a
26 rotating tube lined with refractory material and
27 equipped with a gas burner. The calciner was
28 inclined so that material passed counter current to
29 the gas flow and passed through a hot zone before
30 being discharged into a receiving tray. The final
31 processed material was hard to the touch and light
32 brown with a speckled surface in appearance. The

1 bulk density of the product varied from 560 kg/m³ to
2 920 kg/m³ by adjusting the feed rate and speed of
3 rotation of the calciner.

4

5

6 Example 3

7

8 1,130 kg of the final processed material was further
9 treated to reduce its particle size by milling
10 whereby it was passed it through a rotating cylinder
11 containing heavy balls of steel such that the
12 tumbling action of the balls crushed the final
13 processed material to a particle size of less than
14 100 µm. The milled material was tested by
15 incorporating it in the formulation of concrete
16 products, such as concrete blocks for building. In
17 tests a substantial part of the cement content of
18 the formulation was replaced by the milled material
19 which produced satisfactory concrete blocks having
20 sufficiently similar properties to existing 'breeze
21 blocks' so as to be direct substitute therefor.

22

23 The present invention provides an eco-friendly
24 method of using a significant waste product, that is
25 currently simply deposited in landfill sites or
26 spread on land. The process produces a material
27 which is usable in a number of industrial
28 applications, thereby not only increasing the
29 recyclability of waste papers, but provided a
30 beneficial product.

31